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RESEARCH ARTICLE

Functional and nutritional properties of infrared- and microwave heat-moisture-treated sorghum meals

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Abstract

Background and Objectives: Starch modification using heat-moisture treatment (HMT) has been proven to influence starch functionality and nutritional properties as it can increase resistant starch content and reduce the glycemic index (GI). This study aims to determine the nutritional and functional properties of infrared (IR)- and microwave (MW) heat-moisture-treated (HMT) white and red non-tannin, and red tannin sorghum meals with the aim of further reducing the estimated GI.

Findings: All treated meals had significantly ($p < .05$) lower pasting peak viscosity than the untreated samples, possibly due to aggregate formation observed under the light microscope, restricting the starch from swelling to form a high-viscosity paste. There was a decrease in the percentage of starch hydrolysis between the sorghum types and a further decrease after HMT treatment. A significant difference was observed in the protein digestibility between the sorghum types, but not between the treatments.

Conclusion: HMT with IR and MW further reduced the starch digestibility, possibly because of changes in the starch molecular configuration.

Significance and Novelty: This study suggests that the changes in the starch functionality and nutritional properties of the HMT-treated sorghum meals can potentially be useful in the development of lower-GI sorghum foods. The results also differentiate the characteristics of sorghum types.

KEYWORDS

estimated glycemic index, heat-moisture treatment, infrared, microwave, resistant starch, tannin and nontannin sorghum meal

1 | INTRODUCTION

Starch is the main component of sorghum grain, ranging from 68% to 75% (Souilah et al., 2014). The glycemic index (GI), which is a percentage of the incremental glucose area under the curve of a test item that is compared with a standard food, can be used to determine the digestibility of a product that is high in carbohydrates (Willett et al., 2002). Starch can be classified as slowly digestible starch (SDS), rapidly digestible starch (RDS), and resistant starch (RS)

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based on the hydrolysis rate (Englyst et al., 1992). Slowly digesting starches have the advantage of releasing blood glucose gradually, which can help with satiety and lower the glycemic response, both of which can lower the risk of metabolic syndromes such as obesity, type 2 diabetes, and cardiovascular disease (Adebo, 2020). As a dietary fiber, RS can aid in the regulation of blood sugar, cholesterol levels, and mineral absorption (Raigond et al., 2015).

Compared to other cereals, sorghum grain is known to be more slowly digested due to its hard peripheral endosperm layer, strong interaction between endosperm proteins (kafirins) and starch granules, and tannins that prevent α-amylase from accessing starch (Barros et al., 2012; Taylor & Duodu, 2023). However, digestibility can only be determined on a processed product, and the processing conditions used in a sorghum-based product will determine the rate of starch hydrolysis. Despite the statements in the literature that "sorghum foods are slowly digested starch and good for people with diabetes and obesity" (Rashwan et al., 2021), other researchers have shown sorghum foods to have an intermediate and high GI (Moraes et al., 2015). An in vitro starch digestibility study carried out by Moraes et al. (2015) reported the estimated GI for decorticated sorghum and pasted whole-sorghum flour to be 84.52 and 68.26, respectively. This was also similar to another study in the literature of nontannin sorghum flours, which ranged in GI between 63.19 and 83.36 (Souilah et al., 2014). However, it is essential not to overlook the GI and glycemic load of sorghum-based foods after processing. The GI and glycemic load are increased when starchy foods are heated to a point where starch gelatinizes, losing its crystallinity and generating a disordered structure that is more susceptible to enzymatic hydrolysis (Jung et al., 2009). A study by Nathakattur Saravanabavan et al. (2013) reported a GI of popped sorghum starch between 85 and 92. Thus, there is a need to modify sorghum starch to reduce its GI further.

Heat-moisture-treated (HMT) is the physical modification of starch involving a low moisture level (10%–30%) and heating temperatures (90–120°C) above the glass transition temperature but below the gelatinization temperature. This modification changes the physicochemical properties of starch without destroying the granular structure (Zavareze & Dias, 2011). The gelatinization transition temperature increases as a result of HMT, granular swelling and amylose leaching decrease, and thermal stability increases. The modification of starch by HMT has been carried out using microwave (MW) (Deka & Sit, 2016) and infrared (IR) energy (Mapengo & Emmambux, 2020) on taro and maize starch, respectively. There is limited research on the use of HMT on sorghum meals using MW and IR energy with the aim of further reducing the estimated GI. This study aims to determine the effects of HMT using MW and IR energy on the functional and nutritional properties of type I white and red non-tannin and type III red tannin sorghum meal.

2 | MATERIALS AND METHODOLOGY

2.1 | Materials and raw material characterization

Three types of sorghum grains, type I white non-tannin sorghum (Macia) and two red hybrid cultivars namely, type I red non-tannin known as GM and type III red tannin known as GH, which are commercially produced in South Africa, were used. All other chemicals were of analytical grade. Whole-sorghum grains were cleaned and milled using a laboratory hammer mill (Perten Lab mill 3100; PerkinElmer) with a 1 mm sieve size. The meal samples were analyzed for moisture, ash, and crude fat using procedures 925.10, 923.03, and 920.39, respectively, of the Association of Official Analytical Chemists (AOAC, 2019). Protein (N × 6.25) was determined using the Dumas combustion method. Tannin content was determined according to Price et al. (1978). In brief, sorghum meal (1 g) was weighed into a 25 mL beaker. Concentrated HCl in methanol (10 mL of 1% v/v) was used for the tannin extraction process by stirring on a magnetic stirrer for 2 h. Vanillin-HCl reagent was added to the extracted tannin and incubated in the dark for 20 min. The absorbance was read at 500 nm using the spectrophotometer (FLUOstar Omega; BMG LABTECH), and the tannin content was expressed as mg Catechin equivalent per 1 g sample. Sorghum grain endosperm texture was estimated according to Taylor and Taylor (2008). It is defined as the proportion of corneous (horny/glassy/steely/vitreous) endosperm relative to floury (milky/opaque/chalky) endosperm.

2.2 | Heat-moisture treatment by IR and MW energy

The moisture level of the different types of sorghum meal was equilibrated at 25%, as described by Mapengo and Emmambux (2020). The equilibrated samples were heat-moisture-treated by IR and MW energy at 250 W for 15 min. A preliminary test showed a burn in the samples after 20 min; therefore, a 15 min treatment duration was chosen. IR and MW heating was performed using an IR/MW Hot air tunnel oven (MW180; Delphuis Technologies) with four halogen lamps (0.2–4 μm). Sorghum meal

(20 g) was placed in glass Petri dishes with a thickness of 0.31 cm. The height of the sample away from the IR lamps was 10 cm. An IR thermometer was used to detect the surface temperature of the samples, which was about 100°C. Heat-treated samples were cooled at room temperature and kept in the oven at 40°C for 12 h to obtain a uniform moisture content of about 7%. The dried sorghum meal samples were stored in airtight bags and placed in an air-tight container at 4°C before analysis.

2.3 | Light microscopy

The micrographs of the starch granules were observed using light microscopy. About 10 mg (dry basis) of HMT and pasted sorghum meal was dispersed in 1 ml of 30% glycerol solution and vortexed. Two (2) drops of the suspension were placed on a slide and covered with a coverslip. A Nikon Optiphot Transmitted Light Microscope equipped with phase-contrast optics was used to observe the samples. Polarized light was used to visualize birefringence in the granules, and iodine was used to stain the starch granules.

2.4 | Differential scanning calorimetry (DSC)

The thermal properties of the HMT sorghum meal before and after pasting were determined according to a method described by Wokadala et al. (2012) using a high-pressure DSC system (HP DSC827e; Mettler Toledo). To create a homogeneous slurry, 10 mg of sorghum meal was weighed and approximately 30 mg of distilled water was added, resulting in a 1:3 (w/w) starch-to-water ratio. The sealed pans were left at 25°C for a day to reach equilibrium. Samples were scanned from 25°C to 140°C at a rate of 10°/min. The reference was an empty aluminum pan. Indium (Tp = 156.6°C, 28.5 Jg^{-1}) was the standard that was applied.

2.5 | X-ray diffraction (XRD) crystallinity

XRD was conducted using PANalytical X'pert PRO on native, HMT-treated, and pasted samples as described by Mapengo et al. (2019), with slight modifications. Using water, the meal samples were brought to room temperature and allowed to equilibrate for 3 days at a 95% estimated relative humidity. The XRD operating conditions were X-ray diffraction with a Cu Kα ($\lambda = 0.154$ nm) radiation source. Scanning was carried out from 5° to 90° (2θ) with an exposure time of 16 min 14 s, step size of 0.026°, and a time/step ratio of 229.5 s. Origin Pro 16 software® was used to plot graphs and calculate the relative crystallinity (RC). The RC was the area of crystalline peaks divided by the area of all peaks multiplied by 100.

2.6 | Meal pasting properties

Sorghum meal samples were pasted according to Wokadala et al. (2012), with modifications involving the use of a rheometer with a starch cell (Physica MCR 101). The treated and untreated sorghum meals were pasted with an initial stirring of 960 rpm at 50°C for 10 s. Within 2 min, stirring was performed at 160 rpm and heated to 91°C at a rate of 5.5°C/min. The samples were then held at 91°C for 15 min, followed by cooling from 91°C to 50°C at a rate of 5.5°C/min, and allowed to stand for 2 min.

2.7 | Water absorption and solubility index

Sorghum meal solubility was determined using the method described by Mapengo and Emmambux (2020). Samples of (1 g, dry basis) meal were cooked in 10 mL of distilled water at two different temperatures, 50°C and 91°C, in a shaking water bath at 150 rpm for 30 min, with the mixture being vortexed every 5 min. The sample solution was centrifuged at 9154.3*g* for 15 min. In an air oven, the supernatant was decanted and evaporated at 105°C for 16 h. The solubility index was determined as the ratio of the dried supernatant's weight to the dry meal's weight and expressed as a percentage (%). After centrifugation, the residue was weighed to determine the absorption index. A pellet's weight (g) per gram of dry ground material was used to express the water absorption index (WAI).

2.8 | Nitrogen solubility index (NSI)

The NSI was determined according to Ogundele et al. (2017). Meals (1 g) were mixed for 1 h at 30°C in 20 mL of a 0.1 M NaCl solution. After centrifugation (9154.3*g*, 15 min), the supernatant was filtered using Whatman No. 1 filter paper. The residue from the suspension was washed two more times in 10 mL of 0.1 M NaCl solution at pH 7. After freezing at −10°C overnight, the filtrate was freeze-dried using a 13KL Instruvac Lyophilizer. A Dumatherm (DT; Gerhardt Konigswinter) was used to measure the amount of nitrogen present in the freeze-dried sample. The total

nitrogen content of the freeze-dried sample divided by the total nitrogen content of a flour sample on a dry basis was the formula used to calculate the NSI.

2.9 | Fourier-transform infrared (FTIR) spectroscopy

FTIR spectroscopy was performed using a Bruker FTIR spectrometer, on native, HMT-treated, and pasted samples in the 400–4000 cm^{-1} range. The OPUS operation system software included in the FTIR spectrometer was used to obtain the spectra. To serve as a control, the background was scanned before the sample analysis. Using a spatula, the sample was placed on the attenuated total reflectance (ATR) crystal to cover the diamond surface. FTIR spectra were obtained at a resolution of 4 cm^{-1} and 32 scans. Deconvolution of the amide group ranging from 1600 to 1700 cm^{-1} was isolated from the whole spectra using Origin Pro software®. Baseline correction was performed. The α-helix (1650–1658 cm^{-1}) and β-sheet (1620–1640 cm^{-1}) peaks were identified from the second derivative spectrum. Peak fitting was performed using the Gauss curve fit.

2.10 | In vitro starch digestibility

In vitro starch digestibility of the pasted sorghum meals was determined using the method described by Goñi et al. (1997). In a conical flask, 50 mg of the material was weighed. First, 0.2 mL of a solution containing 1 g of pepsin (Sigma-Aldrich P7000-100G) in 10 mL of HCl-KCl buffer (pH 1.5) was added. Samples were incubated in a shaking water bath at 40°C for 60 min and made up to 25 ml volume with tri-maleate buffer (pH 6.9). Five milliliters of Tris-maleate buffer containing 2.6 IU of porcine pancreatic α-amylase was added, and the samples were incubated in a shaking water bath at 37°C. Aliquots (1 mL) were taken at time points of 20, 60, 90, 120, and 180 min. Aliquots were boiled at 100°C for 5 min to inactivate the enzyme and refrigerated until the end of the incubation period. To each aliquot, 3 mL of sodium acetate buffer (pH 4.75) and 60 μL of amyloglucosidase (3260 U/mL) were added, and subjected to 45 min of water bath incubation at 60°C. The volume was adjusted to 10 mL. Using GOPOD reagents, the amount of glucose released during starch hydrolysis was determined. According to (Englyst et al., 1992), starch fractions were calculated as follows:

$$\text{Rapidly digestible starch (RDS)} = \text{Glucose released within 20 min (G20)} \times 0.9,$$

$$\text{Slowly digestible starch (SDS)} = \text{Glucose released within 120 min (G120)} - \text{G20} \times 0.9,$$

$$\text{Resistant starch (RS)} = (100\text{-G120}) \times 0.9.$$

2.11 | In vitro protein digestibility

In vitro protein digestibility was determined according to the pepsin method described by Hamaker et al. (1986), with modifications. The pasted meal (200 mg) was mixed in 5 mL of citrate buffer (pH 2) and vortexed to ensure complete dispersion. Citrate buffer containing pepsin (28 mL, 131 mg pepsin/100 mL buffer) was added and vortexed. Samples were incubated in a shaking bath at 37°C for 2 h.

The addition of 2 mL of 2 M sodium hydroxide was used to stop the digestion process. The samples were centrifuged for 10 min at 9154.3*g*. After discarding the supernatant, the residue was subjected to two centrifugation cycles by washing with 30 mL of distilled water. The residue was dried in an air oven overnight at 100°C. The Dumas combustion method was used to determine the protein content of the dried residue. In vitro protein digestibility was calculated as the initial total weight of protein minus the residual weight divided by the initial total weight of protein, multiplied by 100.

2.12 | Statistical analysis

Data were analyzed with IBM SPSS® statistics using a multivariant general linear model based on a 95% confidence level. The significant difference between means of the values obtained from the different sorghum types and treatments was calculated using the Tukey honestly significant difference (HSD) test with a $p \leq .05$. All the experiments were repeated three times. The means of the analysis were subjected to principal component analysis (PCA) using Origin Pro® software.

3 | RESULTS AND DISCUSSION

3.1 | Proximate composition of sorghum meals

The proximate composition, total starch, and tannin content of the sorghum types are shown in Supporting Information: Table S1. One red-type sorghum contained a tannin content of 1.64%, which was negligible, and

there was no tannin content in the other red and white sorghum types. There was a significant difference ($p < .05$) in the ash, protein, fiber, and starch contents between the three sorghum types, with red tannin showing the highest ash, fiber, and protein contents. Starch, the main component of sorghum, ranged from 72.44 to 76.54% among the sorghum types.

3.2 | Effect of HMT on the thermal properties and XRD of sorghum meals

The heat flow was determined for the native sorghum types and after HMT using the DSC. Their thermal parameters such as onset temperature T_o, conclusion temperature T_c, peak temperature T_p, and enthalpy of change (ΔH) are presented in Table 1. All sorghum types showed the first endotherm ranging from 67.75°C to 84.55°C. This endotherm is likely to be that of starch gelatinization (Parniakov et al., 2018), but the contribution of protein denaturation should not be ignored (Rodriguez Furlán et al., 2012). A significant difference ($p < .05$) was found for T_o and T_c between sorghum types, but no difference ($p > .05$) was found for the peak temperature and the enthalpy of change. The T_o and T_c of red tannin type were found to be higher among the sorghum types and progressively increased further in IR-treated samples, followed by MW samples, probably due to conversion into more perfect starch crystallites. According to Ji (2004), perfection of starch crystallites can be measured based on the onset temperature of gelatinization, and higher onset temperatures for gelatinization would result from more perfect crystallites.

During HMT, the crystalline nature undergoes a structural change that increases its thermodynamic stability. The granular strengthening of HMT starches has primarily been attributed to the molecular rearrangement of starch molecules, particularly amylopectin (Lim et al., 2001). However, the change in enthalpy of gelatinization, indicating a more ordered structure of amylopectin and reflecting more energy for the unraveling of double helices during gelatinization, did not support the findings in this study (Gunaratne, 2002). There was a decrease in the change in enthalpy (ΔH) for the HMT samples, but this was not significant ($p > .05$) in relation to the untreated samples. This finding supports the decrease in RC after HMT, as shown in Supporting Information: Table S2. According to Sui et al. (2015), an increase in T_p and a decrease in ΔH could be attributed to more effective crosslinks in the amorphous region and disrupted hydrogen bonds in the crystalline region.

Figure 1 illustrates the X-ray pattern of native sorghum types and HMT-treated samples. With distinctive peaks at 15° and 23° and an unresolved doublet at roughly 17° and 18°, the native sorghum samples showed a typical A-type crystalline polymorph. A-type starches, which are similarly linked to cereals, are distinguished by

TABLE 1 Effects of heat-moisture treatment by microwave and infrared energy on the thermal properties of different types of sorghum meal.

Sample	Treatment	T_o°C	T_c°C	T_p°C	ΔH J/g
White nontannin	Control	67.75 ± 0.23^{f}	79.35 ± 0.06^{cd}	73.60 ± 0.70^{c}	3.93 ± 0.33^{a}
	IR HMT	72.27 ± 1.25^{b}	80.86 ± 0.98^{bc}	74.90 ± 0.30^{bc}	2.72 ± 0.47^{ab}
	MW HMT	69.00 ± 0.33^{def}	79.01 ± 0.28^{d}	74.34 ± 0.42^{bc}	2.12 ± 0.81^{b}
Red nontannin	Control	69.32 ± 0.15^{cdef}	80.49 ± 0.10^{bcd}	73.50 ± 0.41^{c}	3.58 ± 0.12^{ab}
	IR HMT	70.70 ± 0.46^{bcd}	84.55 ± 0.31^{a}	80.37 ± 0.00^{a}	3.79 ± 0.06^{a}
	MW HMT	68.09 ± 0.09^{ef}	79.00 ± 0.42^{d}	73.10 ± 0.38^{c}	3.12 ± 0.89^{ab}
Red tannin	Control	68.77 ± 0.18^{cde}	80.64 ± 0.68^{bc}	74.28 ± 0.51^{bc}	4.02 ± 0.11^{a}
	IR HMT	74.03 ± 0.47^{a}	83.80 ± 0.07^{a}	78.27 ± 3.40^{ab}	3.02 ± 0.29^{ab}
	MW HMT	70.87 ± 0.21^{bc}	81.15 ± 0.06^{b}	75.25 ± 0.45^{bc}	3.58 ± 0.09^{ab}
Sorghum types		*	*	**	**
Treatments		*	*	*	*
Sorghum types × treatments		*	*	*	**

Note: Means within a column with different letters are significantly different ($p < .05$). MANOVA analysis to show significant differences in sorghum types and treatments.

Abbreviations: ΔH, change in enthalpy; HMT, heat-moisture-treated; IR, infrared; MANOVA, multivariate analysis of variance; MW, microwave; T_c, conclusion temperature; T_o, onset temperature; T_p, peak temperature.

*$p < .05$, **$p > .05$.

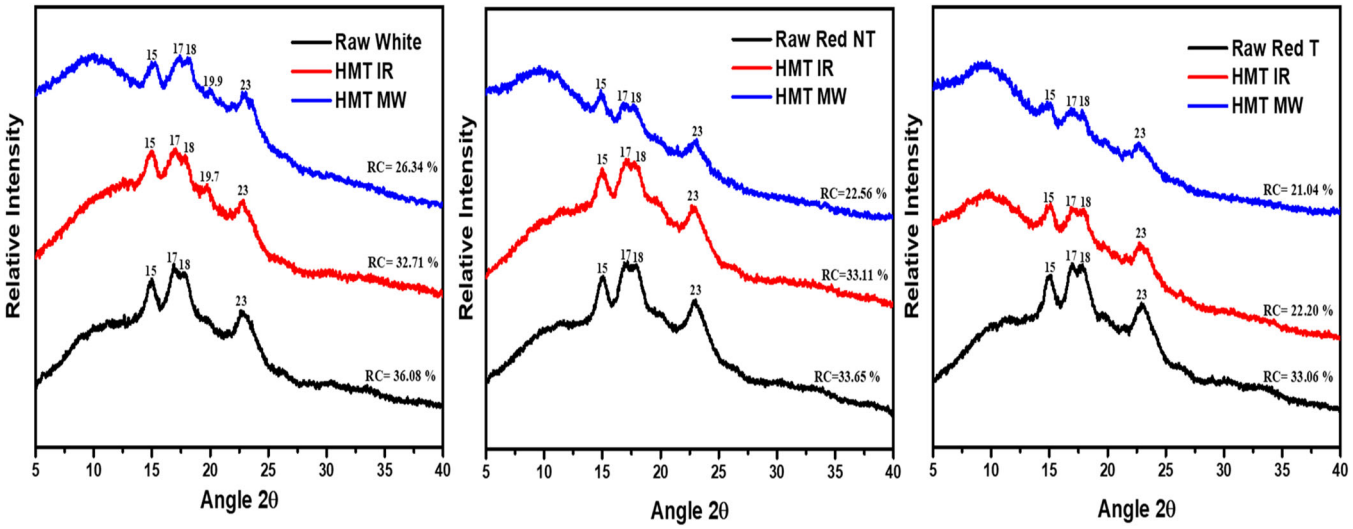

FIGURE 1 Effect of HMT by infrared and microwave treatment on the X-ray pattern on HMT sorghum meals. HMT, heat-moisture-treated; IR, infrared; MW, microwave; RC, relative crystallinity. [Color figure can be viewed at wileyonlinelibrary.com]

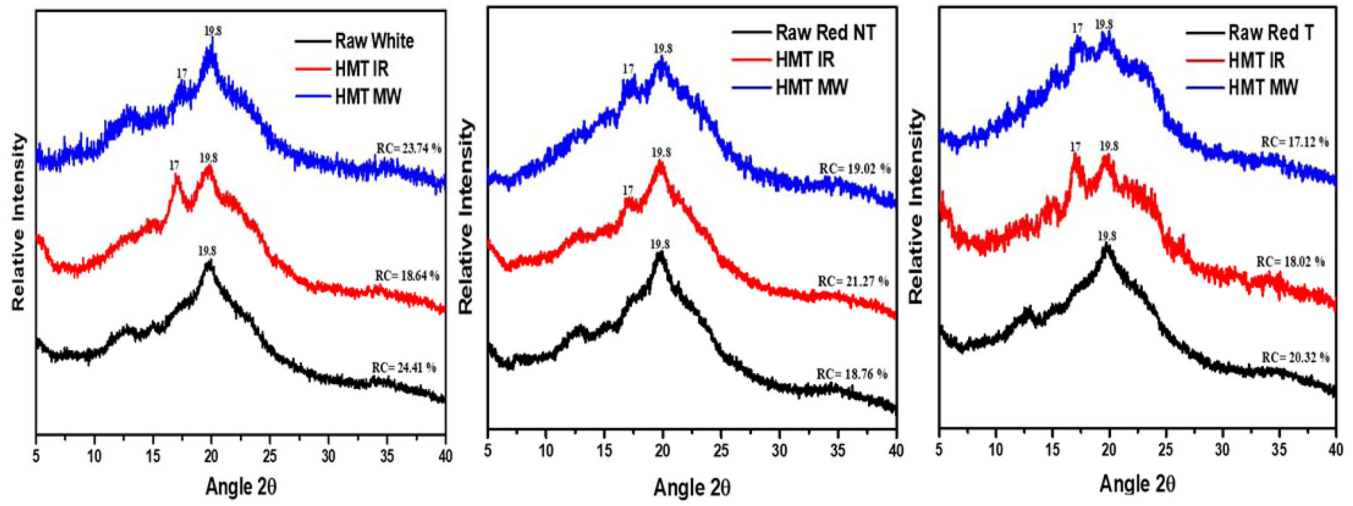

FIGURE 2 Effect of HMT by infrared and microwave treatment on the X-ray pattern on pasted sorghum meals. HMT, heat-moisture-treated; IR, infrared; MW, microwave; RC, relative crystallinity. [Color figure can be viewed at wileyonlinelibrary.com]

strong reflections at 15°, 23°(2Θ) and a doublet at around 17° and 18° (Bhat & Arya, 2020). HMT did not change the characteristic peaks of the native samples. After pasting (Figure 2), the characteristic peak identifying the samples as A-type crystalline polymorphs was not detected. The pasted native raw sample for all the sorghum types showed peaks at 19.9°. Other peaks at 17° and 19.9° were detected in the IR- and MW HMT white and red non-tannin and red tannin sorghum types. Samples showing peaks at about 12.9 and 19.8° are considered V-type crystalline (Sarifudin et al., 2019). This implies that, after pasting, the A-type crystalline pattern partly transformed into a V-type crystalline pattern for all native samples and HMT-treated samples in all sorghum types. However, this finding did not relate to the DSC graph after pasting since no amylose–lipid endotherm was observed (graph not shown).

3.3 | Effect of HMT on pasting properties of sorghum meals

The effects of HMT on the pasting properties of white non-tannin, red non-tannin, and red tannin sorghum are presented in Figure 3. Their corresponding pasting time, pasting temperature, maximum viscosity, and final

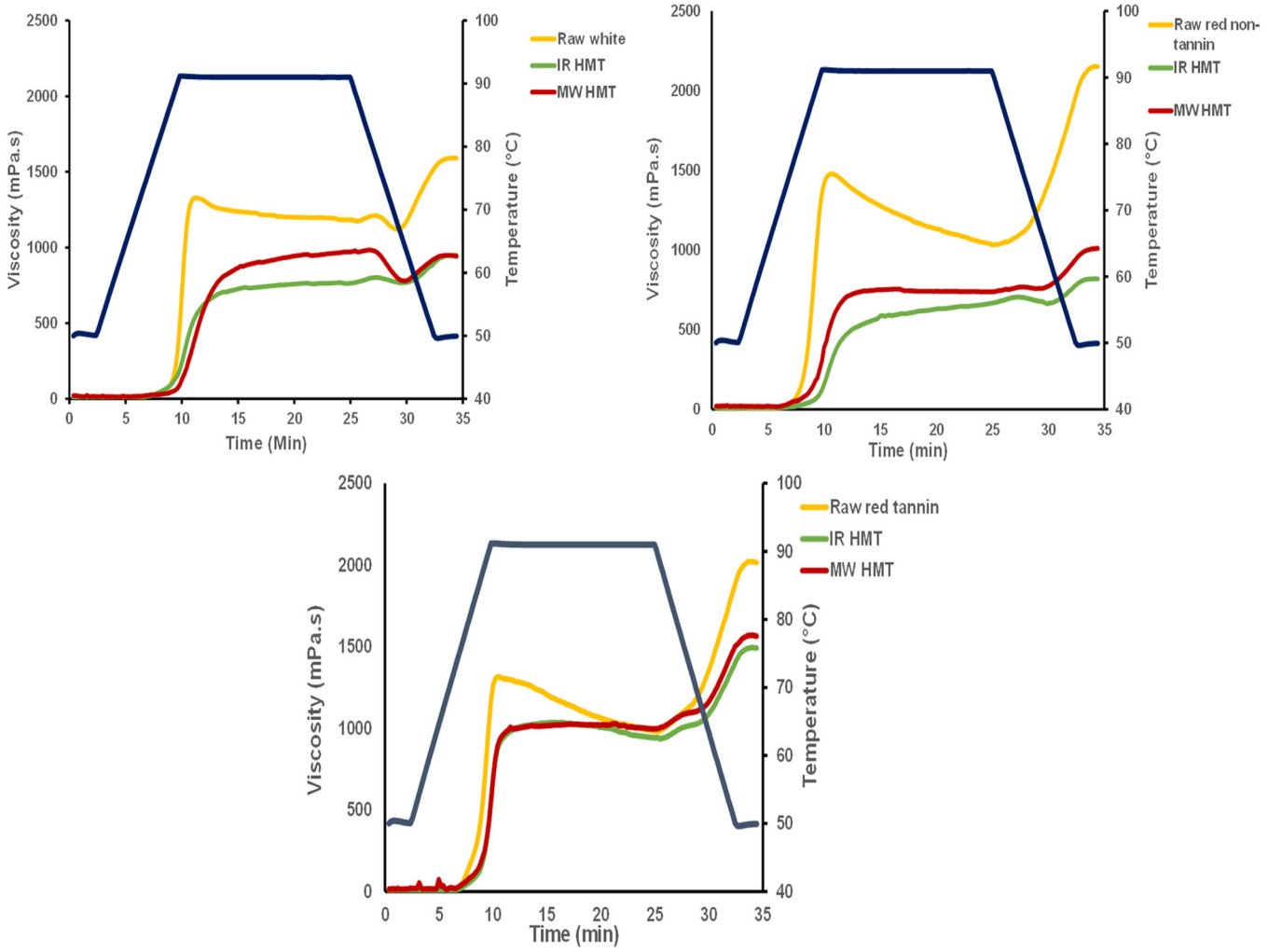

FIGURE 3 Effects of heat-moisture treatment (HMT) by infrared (IR) and microwave (MW) energy on the pasting properties of different sorghum types. [Color figure can be viewed at wileyonlinelibrary.com]

viscosity are presented in Supporting Information: Table S3. There was no significant difference in the pasting time and temperature for any of the sorghum types and treatments. The pasting graph showed that HMT by IR and MW energy decreased the maximum and final viscosities. The reduced maximum viscosity could be due to aggregate formation, as observed in the light micrographs of the HMT samples (Supporting Information: Figures S1, S2, and S3). The aggregate formation could probably be a result of the interaction of starch, protein, and fiber in the meals. This could limit starch swelling to produce lower viscosity. According to Oñate Narciso and Brennan (2018), proteins could prevent the swelling of starch. After treatment, denatured proteins could have formed a protein–starch matrix, thereby strengthening it and limiting its leaching out of starch polymers for higher viscosity. For all the heat-moisture-treated samples, the starch granules were affected by the degree of agglomeration, making them more aggregated. This agrees with the findings of a study carried out by Mapengo and Emmambux (2020), who observed the formation of aggregates of maize starch after HMT by IR energy. It was also observed that the starch granules from the IR-treated samples remained highly birefringent compared to the MW-treated samples, showing minimal gelatinized starch.

3.4 | Effect of HMT on WAI, WSI, and NSI of sorghum meals

The WAI and the water solubility index (WSI) were determined using temperatures of 50°C and 91°C (Table 2). These temperatures were chosen to mimic the temperatures used during pasting. WAI was higher for all sorghum types and treated samples at 91°C compared to samples at 50°C. The higher temperature gelatinized the starch, inducing higher absorption of

TABLE 2 Effects of heat-moisture treatment (HMT) by microwave (MW) and infrared (IR) energy on WAI, WSI, and NSI of different types of sorghum meal.

Sample	Treatment	WAI at 50°C	WSI at 50°C	WAI at 91°C	WSI at 91°C	NSI %
White nontannin	Control	1.21 ± 0.02^{f}	4.84 ± 0.05^{a}	6.86 ± 0.14^{bc}	2.37 ± 0.08^{cd}	62.72 ± 1.54^{a}
	IR HMT	1.87 ± 0.04^{cd}	3.94 ± 0.02^{bc}	5.26 ± 0.14^{e}	4.50 ± 1.72^{abc}	39.04 ± 1.13^{c}
	MW HMT	2.06 ± 0.08^{bc}	4.14 ± 0.30^{b}	5.57 ± 0.38^{de}	2.85 ± 0.32^{bcd}	36.52 ± 1.15^{b}
Red nontannin	Control	1.33 ± 0.03^{f}	4.16 ± 0.36^{b}	8.63 ± 0.18^{a}	1.13 ± 0.08^{d}	61.89 ± 0.84^{a}
	IR HMT	1.86 ± 0.08^{d}	3.39 ± 0.33^{cd}	6.32 ± 0.39^{cd}	6.42 ± 0.32^{a}	32.01 ± 0.87^{e}
	MW HMT	1.98 ± 0.07^{cd}	2.97 ± 0.34^{d}	6.64 ± 0.95^{cd}	5.78 ± 2.39^{a}	25.62 ± 0.81^{d}
Red tannin	Control	1.57 ± 0.14^{e}	2.97 ± 0.05^{d}	7.78 ± 0.27^{ab}	3.80 ± 0.24^{abc}	20.07 ± 0.64^{g}
	IR HMT	2.21 ± 0.02^{ab}	2.77 ± 0.40^{d}	6.41 ± 0.11^{cd}	5.21 ± 0.24^{ab}	17.30 ± 1.37^{g}
	MW HMT	2.38 ± 0.13^{a}	2.88 ± 0.20^{d}	7.10 ± 0.44^{bc}	3.90 ± 0.69^{abc}	16.70 ± 0.27^{f}
Sorghum types		*	*	*	**	*
Treatments		*	*	*	*	*
Sorghum types × treatments		**	*	**	*	*

Note: Means within a column with different letters are significantly different ($p < .05$). MANOVA analysis to show significant differences in sorghum types and treatments.

Abbreviations: MANOVA, multivariate analysis of variance; NSI, nitrogen solubility index; WAI, water absorption index; WSI, water solubility index.

*$p < .05$, **$p > .05$.

water. Treatments significantly affected the WAI and WSI at both temperatures. After HMT, a slight increase in WAI was observed at 50°C, but there was a decrease at 91°C for all sorghum types. The decrease in WAI at 91°C could be an indication of molecular structure changes during HMT as well as aggregate formation resulting in lower pasting viscosity. There was a significant difference ($p < .05$) in the WSI between the sorghum types at 50°C. The white nontannin type showed higher WSI, followed by red non-tannin and red tannin types. The low WSI in the tannin-type sorghum suggests an interaction between the tannin and protein-resisting starch solubility (Dunn et al., 2015).

The NSI of the sorghum meal types, treatments, and interaction between sorghum type and treatment were all significantly different ($p < .05$). The white non-tannin type showed the highest value, followed by the red non-tannin type, and the red tannin type showed the lowest NSI. Given tannin's ability to bind sorghum protein (Emmambux & Taylor, 2003), the complexes formed could have resulted in the lower NSI of the tannin sorghum type. There was a decrease in NSI for all sorghum types after HMT treatment (Table 2), with IR-treated samples showing higher NSI and MW-treated samples showing lower NSI values. The decrease in NSI could have resulted from protein denaturation during HMT. According to Vinay and Sindhu Kanya (2008), heat increases the surface hydrophobicity of protein due to the unfolding of molecules and molecular size effect through hydrophobic interaction and disulfide formation.

3.5 | Effect of HMT on the secondary protein structure of sorghum meals

The FTIR spectra of native white and red non-tannin, and red tannin sorghum meals are shown in Supporting Information: Figure S4. The sorghum types showed similar bands, but the tannin sorghum type showed a band at 1710 cm^{-1}, which could be a result of the tannin content. According to Lin et al. (2021), the phenolic acid band could be identified in sorghum flours at around 1709 cm^{-1}. The absorption bands at around 3700–2500, 1800–1600, and 1200–800 cm^{-1} in the sorghum types indicate that all meals contain hydrogen stretching (C–H, O–H, and N–H), carbonyl stretching vibration C=O, and C–O, C–C stretching, respectively (Smith, 2018). All the sorghum types showed three distinct peaks at 1146–1150, 1007, and 994 cm^{-1}, representing the C–O and C–C vibration of carbohydrates (Smith, 2018). Deconvolution of the Amide 1 group to determine the secondary protein structure was performed on pasted samples in the range 1600–1700 cm^{-1}. The spectra showed an α-helix and a β-sheet (Supporting Information: Figure S5). More proportions of the β-sheet were developed in all samples after HMT.

The development of a β-sheet after HMT results from a noncovalent interaction as a result of denaturation, uncoiling, or unfolding of coiled structures (Yu, 2005). Research has shown the effect of

heat treatment on protein structures, changing the α-helix to β-sheets (Seguchi et al., 2004). This result indicates that the treatment significantly affected the protein structure, which can be seen in the decrease in its protein digestibility, as discussed below.

3.6 | Effect of HMT on in vitro protein digestibility of sorghum meals

The in vitro protein digestibility of pasted native and HMT-treated samples is shown in Table 3. There was a significant difference ($p < .05$) in the protein digestibility between the sorghum types, but there was no significant difference ($p > .05$) between the treatments. Protein digestibility was lower in the red tannin type, followed by red nontannin and white nontannin sorghum type. Tannin in sorghum forms irreversible complexes with protein, which could have resulted in the lower protein digestibility of tannin sorghum (Emmambux & Taylor, 2003). After HMT, there was no significant further change in the protein digestibility of the tannin sorghum type. This was evident in the deconvoluted amide 1 group of the red tannin type (Supporting Information: Figure S5), which showed no obvious change in the β-sheet proportion with the heat-treated samples. There was a decrease in protein digestibility after HMT and pasting, but this was not statistically significant ($p > .05$). Research has found that wet heat treatment generally decreases the in vitro protein digestibility of sorghum flour (Duodu et al., 2002; Hamaker et al., 1986). Pasting following HMT could have induced the formation of enzymatically resistant protein polymers formed by disulfide crossing by β and γ- kafirin. Such cross-linking restricts the digestion of protein (Duodu et al., 2002). Other factors, such as starch, polyphenols, nonstarch polysaccharides, and antinutrients, could have interfered with the protein digestibility of sorghum meals (Duodu et al., 2002).

3.7 | Effect of HMT on starch hydrolysis of sorghum meals

Figure 4 shows the rate of starch hydrolysis of the different sorghum types and their HMT samples. Table 3 shows the derived in vitro data, including the hydrolysis index (HI), the estimated glycemic index (eGI), RDS, SDS, and RS. There was a significant difference between the sorghum types and the treatments. Based on the literature, tannin restricts the digestion of starch, and this was observed in the significant decrease in the HI between non-tannin and tannin sorghum types. A significant decrease was also observed after HMT, and subsequently, a decrease in the eGI was observed. With the significant

TABLE 3 Effects of heat-moisture treatment (HMT) by microwave (MW) and infrared (IR) energy on the in vitro starch and protein digestibility of different types of sorghum meal.

Sample	Treatment	HI	eGI	SDS	RDS	RS	Protein digestibility %
White nontannin	Control	85.23 ± 0.79^{a}	86.50 ± 0.43^{a}	19.48 ± 3.66^{ab}	72.83 ± 3.12^{a}	7.69 ± 1.62g	61.39 ± 1.18^{a}
	IR HMT	72.61 ± 0.40^{d}	79.57 ± 0.22^{d}	14.42 ± 0.70^{bc}	64.23 ± 0.33^{b}	21.34 ± 1.01^{e}	57.95 ± 0.11^{a}
	MW HMT	77.70 ± 0.93^{bc}	82.49 ± 0.80^{bc}	9.36 ± 2.38^{c}	64.38 ± 1.99^{b}	26.26 ± 0.42^{cd}	56.46 ± 0.93^{a}
Red nontannin	Control	79.13 ± 1.19^{b}	83.15 ± 0.65^{b}	2.95 ± 0.58^{d}	73.01 ± 1.15^{a}	24.04 ± 1.52^{de}	41.55 ± 5.54^{c}
	IR HMT	62.34 ± 1.94^{e}	73.94 ± 1.06^{e}	2.58 ± 0.16^{d}	74.33 ± 2.16^{a}	23.09 ± 2.09^{de}	25.67 ± 3.98^{c}
	MW HMT	74.64 ± 0.52^{cd}	80.69 ± 0.29^{cd}	22.30 ± 1.31^{a}	65.65 ± 0.40^{b}	12.05 ± 1.69^{f}	25.12 ± 5.90^{b}
Red tannin	Control	59.94 ± 0.95^{e}	72.62 ± 0.52^{e}	1.82 ± 0.63^{d}	56.44 ± 1.38^{c}	41.74 ± 1.11^{a}	23.78 ± 1.23^{c}
	IR HMT	54.19 ± 1.28^{f}	69.46 ± 0.70^{f}	8.58 ± 1.27^{c}	56.46 ± 0.21^{c}	34.96 ± 1.45^{b}	22.58 ± 2.14^{c}
	MW HMT	54.98 ± 3.51^{f}	69.89 ± 0.3^{f}	10.46 ± 5.21^{c}	59.93 ± 1.93^{c}	29.58 ± 3.69^{c}	17.81 ± 0.33^{c}
Sorghum type		*	*	*	*	*	*
Treatment		*	*	*	*	*	**
Sorghum type × treatment		*	*	*	*	*	*

Note: Means within a column with different letters are significantly different ($p < .05$). MANOVA analysis to show significant differences in sorghum types and treatment.

Abbreviations: eGI, estimated glycemic index; HI, hydrolysis index; MANOVA, multivariate analysis of variance; SDS, slowly digestible starch; RDS, rapidly digestible starch; RS, resistant starch.

*$p < .05$, **$p > .05$.

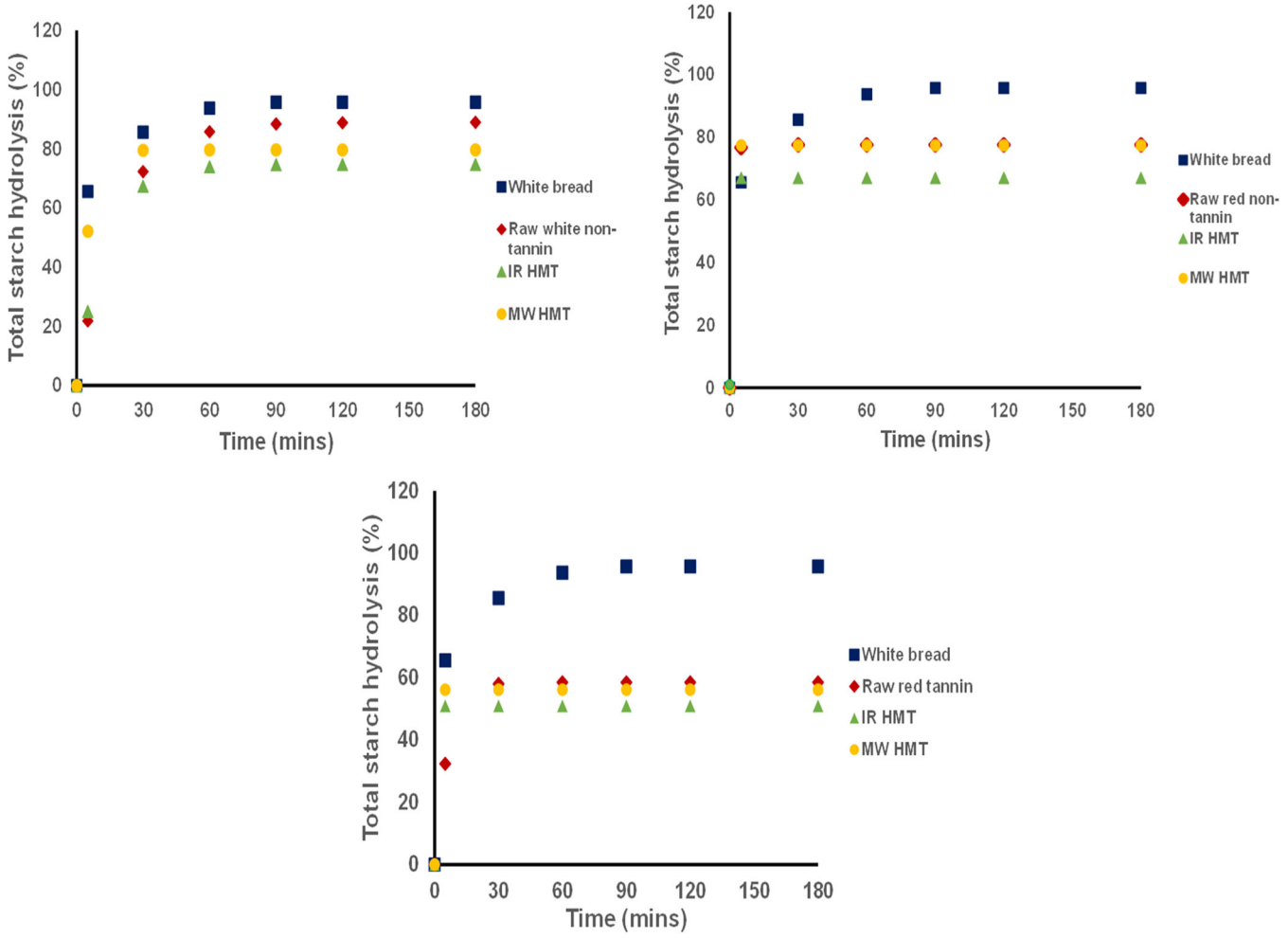

FIGURE 4 Effects of heat-moisture treatment (HMT) by microwave (MW) and infrared (IR) energy on the in vitro starch digestibility of different sorghum types. [Color figure can be viewed at wileyonlinelibrary.com]

decrease in all treated samples, IR-treated samples of each type showed the lowest starch hydrolysis and eGI.

The heat transfer mechanism from IR is radiation heating, which occurs on the surface of the food, and conductive heating, which occurs inside the food. The reduced eGI recorded in IR-treated samples could have been due to starch molecular changes, thus having less free molecular space as a result of more hydrogen bonding (Mapengo et al., 2021). This restrains the movement of the chains, resulting in an increase of SDS and subsequently reducing the eGI. The fact that the tannin sorghum types showed lower GI and higher RS content compared to the non-tannin types may be attributed to the presence of tannin. The interactions of tannin in sorghum with digestive enzymes and protein have been reported to reduce nutrient digestibility. According to Barros et al. (2012), tannin can be adsorbed on starch granules and act as α-amylase inhibitors, hence the observed increase in RS content.

3.8 | PCA of sorghum meals and effects of HMT

PCA was carried out to understand the correlation between sorghum types and heat-moisture treatments, which explained 73.52% of the variations (Figure 5). The first principal component (PC1) explains 44.16% of the variations in the nutritional properties of sorghum meals in relation to sorghum type and heat-moisture treatment. Heat-moisture treatment by IR or MW decreased the in vitro starch and protein digestibility of sorghum compared to the control and this decrease is independent of the sorghum type. This was clear as the HMT samples have a positive correlation with high RS content, while control samples have a positive correlation with high HI, RDS, and GI. This suggests that there was a change from RDS to SDS for MW- and IR-treated white and red non-tannin sorghum types. The cluster of MW- and IR-treated white and red non-tannin

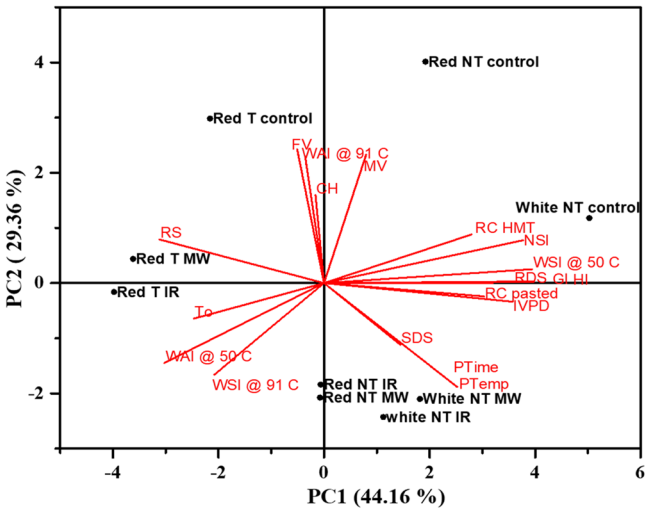

FIGURE 5 Principal component analysis biplot for loading of measurable and scores of sorghum types and heat-moisture treatment by microwave and infrared energy. CH, change in enthalpy; FV, final viscosity; GI, glycemic index; HI, hydrolysis index; IVPD, in vitro protein digestibility; MV, maximum viscosity; NSI, nitrogen solubility index; PTime, pasting time; PTemp, pasting temperature; RS, resistant starch; T_o, onset temperature; WAI @ 50, water absorption index at 91°C; WAI @ 91, water absorption index at 91°C; WSI @ 50, water solubility index at 50°C; WSI @ 91, water solubility index at 91°C; RC HMT, relative crystallinity of HMT sample; RC pasted, relative crystallinity of pasted samples; RDS, rapidly digestible starch; SDS, slowly digestible starch. [Color figure can be viewed at wileyonlinelibrary.com]

sorghum types had a high content of SDS compared with the other samples. Irrespective of the heat treatments, the tannin sorghum type showed significantly higher RS content compared with the non-tannin sorghum types (Table 3).

The second principal component (PC2) describes the functional properties of the sorghum meals and explains 29.36% of the total variation in relation to the heat-moisture-treated samples and the control samples. Independent of sorghum type, heat-moisture-treated sorghum meals had a lower WAI at 91°C, and maximum and final viscosity than control samples. A high onset temperature, WAI at 50°C, and WSI at 91°C clustered with IR-treated red tannin sorghum meals. Also, red tannin control meal had a strong positive correlation with high final viscosity, WAI at 91°C, RS content, and change in enthalpy.

This supports the findings that the non-tannin sorghum types had a high starch HI, leading to higher RDS content and subsequently higher eGI due to lower RS content compared with high tannin sorghum. This PCA allowed the separation of the effect of sorghum types and HMT on nutritional and functional properties.

4 | CONCLUSIONS

Heat-moisture treatment by IR and MW energy of sorghum meals can affect the pasting properties by lowering the peak viscosity due to aggregate formation observed in the light micrographs, limiting starch swelling. The in vitro starch and protein digestibility of sorghum was more affected in the tannin-type sorghum than the heat-moisture treatment. Sorghum tannin reduces nutrient digestibility by interacting with digestive enzyme and proteins. Protein–tannin interaction induces a barrier around the more crystalline starch granules formed, and phenolic enzyme inhibition slows down the α-amylase enzyme attack on the starch. This led to an increase in SDS and subsequently lower estimated GI. Denatured proteins and tannin–protein interaction led to lower nitrogen solubility and in vitro protein digestibility.

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SUPPORTING INFORMATION

Additional supporting information can be found online in the Supporting Information section at the end of this article.

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